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The use of field dependence of AC susceptibility for the interpretation of magnetic mineralogy and magnetic fabrics in the HSDP-2 basalts, Hawaii

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Abstract

We applied the field dependence parameter χ_{Hd} (%)=[($k_{300\ A/m}-k_{30\ A/m}$)/$k_{300\ A/m}$]×100 given by de Wall for the subaerial and submarine basalts drilled by the 3109 m deep HSDP-2 borehole on Hawaii in order to verify the hypothesis that mainly composition controls the field dependence of AC susceptibility in titanomagnetite of natural occurrences. When we used this parameter, our data showed a significant scattering compared to data presented in earlier studies. In addition to composition, the effect of measurement temperature, grain size and anisotropy on the field dependent susceptibility were examined and found to be critical. The impact of grain size is weaker than the other effects. It cannot be totally excluded that the observed effects arise indirectly through an overlap of the other effects for the investigated basalts. The most important factor for the variation of field dependence is the degree of oxidation, causing a modification of the titanomagnetite composition or formation of titanomaghemite, and the mixing of Ti-rich with Ti-poor titanomagnetites, which strongly reduces the χ_{Hd} parameter. Field dependence is not only related to titanomagnetite composition, especially for intermediate titanomagnetites with T_Cs between 100 and 300 °C. Temperature dependent susceptibility measurements at different field amplitudes for these intermediate types showed at constant geometry of the $k(T)$ curve great differences in susceptibility, resulting in significant changes of the field dependence parameter over the temperature interval from −100 to 260 °C. Therefore variations of the ambient measurement temperatures are able to influence the field dependence. The second important effect is the degree of particle shape and alignment, which controls the field dependence in different orientations especially for the intermediate titanomagnetite, which is intensively intergrown with elongated hemoilmenite grains. As a consequence, samples with higher degrees of anisotropy exhibit differences of the field dependence parameter if measured parallel to k_{max} or k_{min} axis. Therefore, in addition to compositional effects and the temperature dependence, the magnetic fabric has to be considered for the interpretation of field dependent susceptibility measurements. The influence of intrinsic (Ti-content, magnetocrystalline anisotropy), and extrinsic (shape and alignment of grains) factors for the interpretation of the

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degree of anisotropy has to be kept in mind when interpreting AMS data in terms of strain rates experienced by moving lava during emplacement.

Keywords: magnetic susceptibility; titanomagnetite; field dependence; magneto-mineralogy; magnetic fabric; basalt; HSDP

1. Introduction

The low-field magnetic susceptibility is one of the most important magnetic properties in geological applications and is measured as a function of different parameters. Bulk magnetic susceptibility (k) helps to identify dia-, para- and ferrimagnetic behavior of minerals and rocks. The anisotropy of magnetic susceptibility (AMS) determines magnetic fabrics that can be correlated with tectonic fabrics or magmatic flow fabrics. The temperature dependence of magnetic susceptibility ($k(T)$) is applied to identify magnetic minerals. The magnetic susceptibility depends in a complex way on measurement parameters like frequency (e.g. [1,2]) and field amplitude (e.g. [3,4]) but also on grain size and composition. Worm et al. [3] described for coarse-grained ferrimagnetic pyrrhotite, and Kletetschka and Wasilewski [5] for multidomain hematite (with grain sizes larger than 100 μm) strong field dependence. Studies of Jackson et al. [6] demonstrated that also titanomagnetites, especially those with intermediate compositions, show a field dependence of magnetic susceptibility, and that the field dependence is mainly controlled by composition. These observations on field dependent susceptibility are important for all geological investigations using the magnetic susceptibility, namely the anomaly modeling and magnetic anisotropy or the interpretation of frequency dependence, which can be misinterpreted by an overlapping field dependence, resulting in incorrect interpretations of grain size and compositional parameters.

The relationship between magnetic susceptibility (k) and magnetization (M) is given by the Rayleigh law: $M=kH+\alpha H^2$, where k is the initial susceptibility, H the applied field and α the Raleigh coefficient ($\alpha=ck^2$, c is the proportional constant). In small fields (ca. <200 A/m, [6]) the quadratic term is negligible and a linear relation exists between susceptibility and magnetization. At higher fields, the energy is high enough to move the domain walls, which causes a non-linear behavior. In general, each material in a magnetic field creates a field opposite to the external field, called self-demagnetization, which depends on the magnetostatic energy and the wall displacement energy (magnetostriction). The susceptibility of pure magnetite is equal to the self-demagnetizing limit of 3 SI for a sphere and $<1/N$ for Ti-bearing titanomagnetite. It is controlled by the intrinsic susceptibility and the self-demagnetizing factor N according to $k_e=k_i/(1+Nk_i)$, where k_e and k_i refer to extrinsic (susceptibility that is measured by most instruments) and intrinsic susceptibility. The intrinsic susceptibility varies in approximate proportion with the quadratic term of the saturation magnetization, which is as a function of the Ti-content in titanomagnetite and decreases with rising Ti-contents. A principal feature of magnetic properties is its anisotropy. The magnetocrystalline anisotropy, which describes the energy necessary to rotate magnetization direction from the easiest spontaneous magnetization direction (111-direction in titanomagnetite) into the field direction, increases when concentrations of the highly anisotropic Fe^{2+}-ion increases in intermediate titanomagnetite compositions [7].

Because the field dependence of magnetic susceptibility in titanomagnetite is mainly controlled by composition, de Wall [8] proposed a field dependence parameter χ_{Hd} $(\%)=[(k_{300A/m}-k_{30A/m})/k_{300A/m}]\times 100$, similar to the frequency dependence parameter given by Dearing et al. [1] and Muxworthy [9]. De Wall [8] showed a linear regression ($R^2=0.97$) between composition (expressed as Curie temperature) and log χ_{Hd} for titanomagnetite-bearing olivine-melilitites from the Riedheim dike, Germany, confirming a strong correlation between field dependence and composition in titanomagnetite (Fig. 1). Additionally to the strong correlation between composition and field dependence, her results have shown that the field has a significant influence on the degree of anisotropy during AMS measurements.

We applied the field dependence parameter given by de Wall [8] for the subaerial and submarine basalts drilled by the 3109 m deep HSDP-2 borehole on

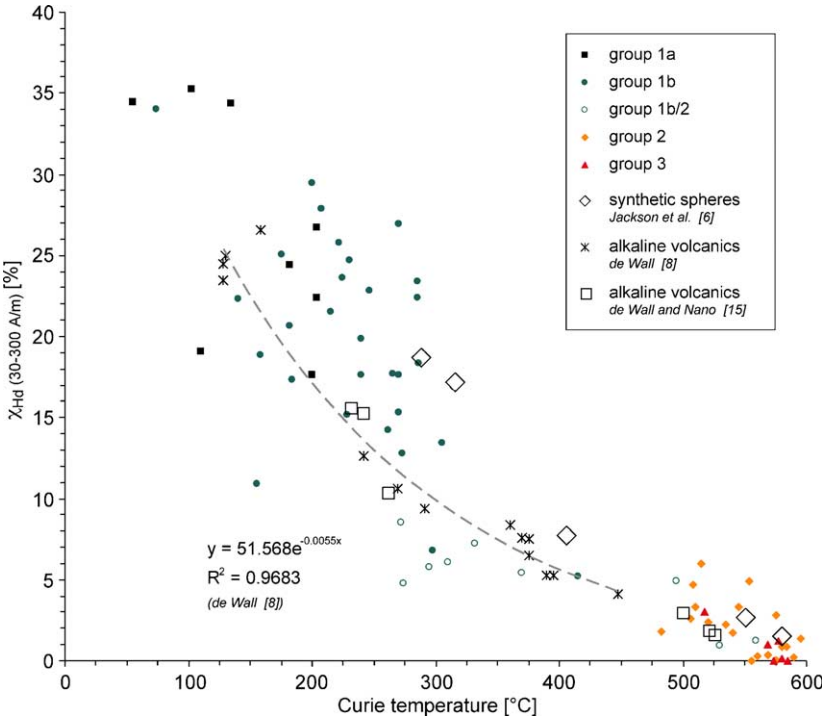

Fig. 1. Field dependence parameter χ_{Hd} calculated from susceptibilities measured at 30 and 300 A/m plotted versus major Curie temperatures for HSDP-2 basalts of different groups (Table 1), mafic alkaline volcanics [8,15] and synthetic titanomagnetite spheres [6]. Dashed line indicates the regression line from de Wall [8].

Hawaii in order to verify the hypothesis that mainly composition controls the field dependence in titanomagnetite of natural occurrences. When we used this parameter, our data showed a significant scattering (Fig. 1) compared to the data shown by de Wall [8]. In order to understand the strong scattering of the field dependence parameter, especially observed for intermediate titanomagnetite compositions, the influence of temperature at which susceptibility is measured, grain size and the arrangement of oxide grains on the field dependence of susceptibility were studied in more detail for the HSDP-2 basalts.

2. Sample material and methods

Susceptibility measurements in different fields were made on subaerial and submarine basalt samples containing titanomagnetite from the 3109 m deep HSDP-2 drill hole in Hawaii. Different groups of magnetic behavior are recognized in the subaerial lava flows related to the degree of high-temperature oxidation during extrusion [10]. Group 1 shows homogenous titanomagnetite of intermediate composition corresponding to Curie temperatures (T_C) between 55 and 305 °C (Table 1), and weak median demagnetizing fields (<20 mT). Further subdivision into 1a and 1b subgroups is based on the low-temperature behavior below ca. −160 °C of magnetic susceptibility, which indicates a higher ferric component in the hemoilmenite phase of group 1b samples. Additionally, samples of this group are characterized by a higher hemoilmenite amount compared to group 1a. The high amount of rhombohedral hemoilmenites is responsible for the predominance of elongated grain textures. Group 2 samples, with exsolution lamellae of hemoilmenite in the titanomagnetites, have higher T_C (480–580 °C), indicating a distinctly higher X_{mt} (magnetite component in titanomagnetite), and higher coercive forces (20–40 mT). Group 3, which shows the highest oxidation stage, is characterized by titanohematite-bearing assemblages with enhanced median demagnetizing

fields (35–85 mT) and a significantly different low-temperature magnetic susceptibility behavior. Mixed types also occur (e.g. 1b/2, 2/1, 3/2), which means that more than one T_C dominates the temperature dependent susceptibility curve. The Fe–Ti oxide assemblage in all basalt samples of this study consists of rhombohedral hemoilmenite and cubic titanomagnetite. Synthetic titanomagnetite–hemoilmenite assemblages, similar in composition to the natural group 1 HSDP-2 samples, were chosen to compare field dependence of intermediate titanomagnetites from natural basalts and synthetic equivalents. Three samples of synthetic multidomain titanomagnetite coexisting with hemoilmenite synthesized at 1100 °C in sub-solidus conditions at 1 bar were provided by D. Lattard and R. Engelmann, Mineralogical Institute, Heidelberg. The X_{ilm} (ilmenite component) in hemoilmenite ranges between 0.90 and 0.92 and the X_{mt} in titanomagnetite between 0.26 and 0.30 (corresponding to T_C between 30 and 160 °C). Further description of the synthetic samples is given by Lattard et al. [12]. Curie temperature (T_C) was determined from temperature dependent magnetic susceptibility measurements ($k(T)$) using a KLY-2 kappabridge under an argon atmosphere at a field of 300 A/m and a frequency of 920 Hz. Determination of T_C was done using the intersection tangent method described in Grommé et al. [13].

The magnetic susceptibility at 30 and 300 A/m was measured for all natural samples using a KLF-3 susceptometer of Geofyzika (now AGICO company) at 2000 Hz and in the field range between 2 and 450 A/m using a KLY-4S kappabridge (AGICO company) at 875 Hz on standard cylinders (2.5 cm in diameter and 2.1 cm in height, with a sample volume of 10.8 cm^3). Directional susceptibility was determined for 192 positions with the KLY-4S and corrections for the diamagnetic sample holder (-2.1×10^{-6} SI) were applied. Calculation of the AMS tensor and the magnitudes of the principle axes of the AMS ellipsoid was done by the measurement program SUFAR (AGICO).

3. Results and interpretation

3.1. Effect of composition

Significant differences in magnetic susceptibility measured in 30 and 300 A/m have been reported for titanomagnetite-bearing olivine-melilitites from a mafic dike (Riedheim, Hegau volcanic province, SW Germany) that contains only cubic spinel phases, titanomagnetite and Cr–Al spinel [8,14]. The field dependence parameter (χ_{Hd}), which has been proposed from these studies, implies a strong correlation between titanomagnetite composition and field dependence in natural samples and allows a rapid detection of titanomagnetite and an estimation of its composition [15]. However the application of this parameter on the HSDP-2 samples revealed a significant scattering of the χ_{Hd} parameter (Fig. 1), especially for intermediate titanomagnetite compositions of groups 1a and 1b. Fig. 2 shows the susceptibility versus field amplitude in the range 2 to 450 A/m for a selected number of samples with T_Cs ranging between 110 and 590 °C. Assuming a proportionality between field dependence and composition, T_Cs should strictly decrease from bottom to top. This is not the case, e.g. for sample SR0456-1.2 or SR0800-0.9, which show a too low susceptibility increase in relation to their composition. The mixed 1b/2 types were not considered in this diagram because different titanomagnetite compositions in one sample result in a “mixed” field dependence parameter (compare Fig. 1 and Table 1).

The composition of titanomagnetite can be estimated from Curie temperature determinations. For correlation with the field dependence parameter, only the major T_C was used. Fig. 3 shows representative $k(T)$ curves for titanomagnetite-bearing HSDP-2 basalts related to groups 1a, 1b, mixed 1b/2, 2 and 3. Part of the group 1a and 1b samples shows a very good reversibility between the heating and cooling run (Fig. 3a and c) demonstrating stable titanomagnetite compositions without alteration features. Other curves show a higher susceptibility in the cooling run, especially in the temperature range above the major T_C, indicating an increase of the phases with the minor, higher T_Cs during the heating experiment. These minor phases are interpreted to represent either stable Ti-poor titanomagnetite compositions crystallized during the lava cooling or titanomaghemites formed by secondary alteration. The occurrence of minor titanomaghemites however, seems not to be critical for the field dependence parameter because some of these samples are within the trend in Fig. 1 while others are not. A deviation from the field dependence–composition trend was also observed for samples with a

Table 1
Curie temperatures, susceptibility measured at 30 and 300 A/m, field dependence parameter calculated from these values, corrected anisotropy factor (from AMS measurements at field amplitudes of 6 and 300 A/m) and grain size ranges of titanomagnetite

Sample	Group	Major T_C [°C]	Minor T_C [°C]	$k_{30\ A/m}$ [10^{-3} SI]	$k_{300\ A/m}$ [%]	χ_{Hd} (30–300 A/m) [%]	P' (6 A/m)	P' (300 A/m)	tmt grain sizes [μm]
SR0131-6.6	1a	204	570	72.6	99.1	26.7		1.034	
SR0132-0.6	1a	55	470	68.8	105.0	34.5		1.083	
SR0132-5.3	1a	102	570	50.4	77.8	35.2	1.039	1.064	20–30, 60–70
SR0799-16.0	1a	135	580	28.6	43.6	34.4	1.015	1.041	30–60, <300
SR0800-0.9	1a	110	213/560	26.7	33.0	19.1	1.023	1.024	10–20, 30–70
SR0944-1.9	1a	182	575	25.4	33.6	24.4		1.097	20–40
SR0951-7.7	1a	200		19.6	23.8	17.6		1.059	20–40
SR0956-15.5	1a	204		39.6	51.0	22.4		1.053	15–40, <130
SR0102-8.9	1b	74	390	10.4	15.8	34.0		1.018	20–60
SR0456-1.2	1b	155	470/590	6.2	6.9	10.9	1.018	1.025	5–10
SR0504-2.3	1b	298	488/580	11.0	11.8	6.8		1.031	1–20, <30
SR0508-4.4	1b	305	485/580	12.9	14.9	13.4	1.025	1.041	
SR0626-11.0	1b	270	520	4.9	5.8	15.5		1.187	
SR0630-4.2	1b	286		6.6	8.1	18.3		1.231	10–20
SR0690-2.7	1b	182	580	5.4	6.8	0.6	1.127	1.182	5–20
SR0691-8.7	1b	175	570	7.0	9.3	25.1	1.132	1.209	15–40
SR0714-18.9	1b	246	490/580	12.5	16.2	22.8		1.240	
SR0715-5.6	1b	240	510	10.1	12.6	19.8	1.159	1.267	10–20, 30–40
SR0716-3.3	1b	200		24.9	35.3	29.5		1.059	
SR0734-4.3	1b	40	576	1.4	1.5	2.1		1.005	
SR0735-10.7	1b	140	580	7.3	9.4	22.3	1.119[a]	1.180	
SR0743-3.4	1b	184	535	6.1	7.4	17.3	1.086[a]	1.144	5–20
SR0743-15.8	1b	208	580	10.1	14.0	27.9	1.080[a]	1.103	10–20
SR0776-18.9	1b	158		10.8	13.3	18.8		1.067	
SR0800-1.0	1b	265	470	19.1	23.2	17.7	1.017[a]	1.026	40–80, <160
SR0800-6.2	1b	215	480	17.9	22.8	21.5	1.013	1.020	
SR0800-19.8	1b	240	505	11.2	13.6	17.6	1.026	1.049	
SR0843-8.5	1b	415	570	12.8	13.5	5.2		1.150	20–50
SR0844-4.4	1b	285	570	6.5	8.4	23.4	1.075[a]	1.120	40–100
SR0844-10.3	1b	270	575	7.1	9.7	26.9	1.041[a]	1.082	30–50
SR0943-2.3	1b	270	570	18.7	22.7	17.6		1.033	
SR0944-0.2	1b	222	576	25.6	34.5	25.8		1.030	<10, 40–70
SR0950-6.7	1b	225		32.3	42.3	23.6		1.058	40–80
SR0951-16.4	1b	230		29.6	39.3	24.7		1.098	40–60
SR0952-1.5	1b	228	485	8.8	10.5	15.6		1.087	
SR0956-2.0	1b	262	480	24.7	28.8	14.2	1.102	1.138	5–35
SR0957-3.9	1b	273	480	28.0	32.1	12.8	1.053	1.101	4–12
SR0959-0.2	1b	285		13.5	17.4	22.4	1.088	1.138	
SR0129-4.5	1b/2	136	550/581	20.5	25.1	18.3	1.013	1.018	20–80
SR0454-4.8	1b/2	370	170	3.0	3.1	5.4		1.028	
SR0501-1.1	1b/2	295	500/585	4.2	4.5	5.8	1.008	1.011	6–30
SR0503-4.8	1b/2	332	518/585	6.2	6.6	7.2		1.006	<5, 10–30
SR0505-8.5	1b/2	274	505/580	3.6	3.8	4.8		1.014	
SR0507-3.2	1b/2	310	505/585	4.2	4.5	6.1		1.015	
SR0836-16.1	1b/2	272	500/575	14.0	15.3	8.5		1.019	
SR0943-2.1	1b/2	495	190/575	11.6	12.2	4.9		1.100	
SR0950-5.8	1b/2	559	256	9.0[b]	9.2[b]	1.4		[b]	
SR0959-4.3	1b/2	530	262	2.1	2.1	1.0	1.027	1.104	
SR0101-7.0	2	584	525	35.7	36.0	0.8		1.029	30–80,<200
SR0131-1.4	2	515	565	65.0	69.1	6.0		1.011	
SR0131-4.4	2	554	467/575	85.6	90.0	4.9	1.021	1.024	<5, 10–30, <50

Table 1 (*continued*)

Sample	Group	Major T_C [°C]	Minor T_C [°C]	$k_{30\ A/m}$ [10^{-3} SI]	$k_{300\ A/m}$ [%]	χ_{Hd} (30–300 A/m) [%]	P' (6 A/m)	P' (300 A/m)	tmt grain sizes [μm]
SR0347-8.9	2	569	425	16.2[b]	16.2[b]	0.3		[b]	
SR0347-10.9	2	482	535	33.5[b]	34.1[b]	1.8		[b]	
SR0348-5.0	2	575	550	12.9[b]	12.9[b]	0.0		[b]	
SR0349-1.0	2	540	340	19.9[b]	20.2[b]	1.7		[b]	
SR0350-0.5	2	545	310/590	10.1[b]	10.5[b]	3.3		[b]	
SR0350-3.4	2	520	570	37.5	38.4	2.3	1.019	1.013	<10, 20–80
SR0352-9.0	2	575	455	7.4	7.4	0.1	1.024	1.045	<5, 10–60
SR0354-1.3	2	535	145/390	15.1	15.4	1.9		1.031	
SR0355-1.0	2	555	505	12.0[b]	12.0[b]	0.0		[b]	
SR0355-2.1	2	510	565	33.7	35.5	5.1		1.004	10–40, <80
SR0355-4.4	2	575	500	10.3	10.5	1.9		1.020	
SR0355-5.7	2	590	430	4.6	4.6	0.2		1.005	
SR0355-10.0	2	560	505	20.5[b]	20.5[b]	0.3		[b]	
SR0652-1.4	2	595		14.1	14.3	1.4		1.004	
SR0715-11.5	2	508	8	22.2	23.3	4.7		1.029	
SR0843-8.1	2	580	165	11.4	11.5	0.9		1.006	
SR0835-19.1	2/1	506	290	10.3[b]	10.6[b]	2.6		[b]	
SR0101-3.0	3	580	520	6.9	6.9	0.1	1.016	1.003	
SR0119-10.8	3	518	564	9.7	10.0	3.0		1.015	
SR0127-0.4	3	569	525	15.4	15.5	1.0	1.016	1.016	
SR0133-4.0	3	573		4.8	4.8	0.0		1.003	10–40
SR0443-6.5	3	585		5.7[b]	5.7[b]	0.0		[b]	
SR0118-0.3	3/2	577	230	2.5	2.5	1.2		1.100	

[a] Measured at 30 A/m.

[b] No standard cylinders, rubbly powder samples, bulk susceptibility has been calculated with basaltic rock densities (average values) ranging from 2.5 to 3.0 g/cm^3 according to Moore [11].

very good reversibility in the $k(T)$ curves, suggesting that there are other factors than composition and mixing of different titanomagnetite compositions in one sample, which influences the field dependence, especially for intermediate titanomagnetites.

Mixed 1b/2 types (Fig. 3e and f) show weak field dependence with χ_{Hd} values below 9% except sample SR0129-4.5 with χ_{Hd} of 18%, indicating a strong influence of the intermediate titanomagnetite with T_C of 136 °C. Samples of this group with major T_Cs ranging between 280 and 400 °C show clearly lower χ_{Hd} with respect to their Curie temperature in Fig. 1. This behavior indicates that in these samples the Ti-poor titanomagnetites with low χ_{Hd} dominate the magnetic behavior, clearly denoting that the mixing of titanomagnetite phases with different composition reduces the field dependence parameter. Some of these samples show irreversible $k(T)$ curves, which are interpreted to reflect titanomaghemite (Fig. 3f). Maghemitization leads to decreasing Fe^{2+} content in the Fe–Ti oxide and therefore a decrease in magneto-crystalline anisotropy [7]. This process reduces the amplitude of the wall displacement energy barriers, which results in lower field dependence.

In general samples from groups 2 and 3 (Fig. 3g and h) show high Curie temperatures, and only weak field dependence (Figs. 1 and 2). This is in accordance with a high-temperature deuteric oxidation of the primary titanomagnetite creating Ti-poor titanomagnetite and oxyexsolutions of hemoilmenite [10]. Scattering of group 2 and 3 data is much less compared to the titanomagnetite with intermediate compositions.

3.2. Effect of temperature on the field dependence of intermediate titanomagnetite

We have observed that especially titanomagnetites of intermediate composition (groups 1a and 1b) show a strong scattering of the χ_{Hd} parameter, for which magnetic susceptibilities were measured at room temperature. The temperature dependent magnetic susceptibility curves for these samples (Fig. 3) show a

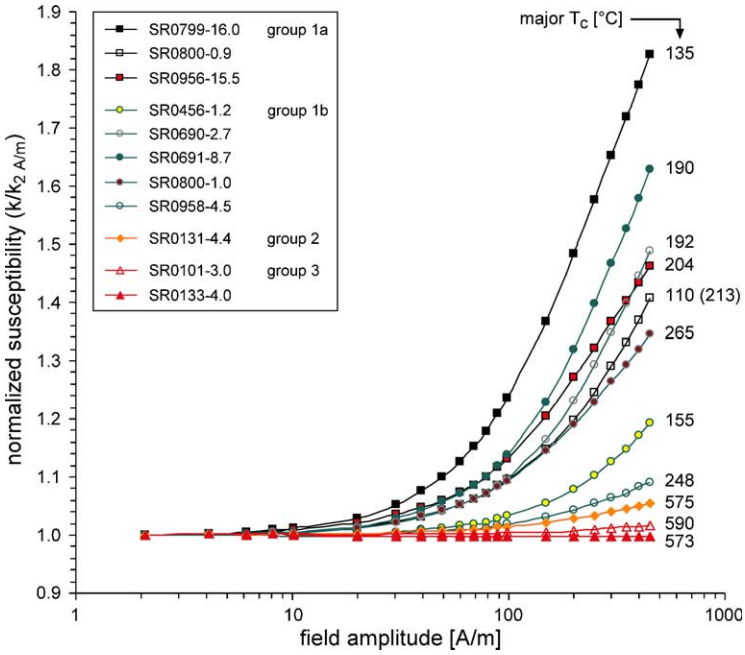

Fig. 2. Magnetic susceptibility (normalized to value at 2 A/m) as a function of field amplitude. The numbers give the titanomagnetite Curie temperature (T_C). The increase in susceptibility begins for the different samples at different fields.

pronounced increase of magnetic susceptibility before reaching the T_C, which already occurs around room temperature. Below T_C the temperature dependence of magnetic susceptibility depends on the relationship of $M_S(T)/K(T)$, where M_S is the spontaneous magnetization and K is the magnetocrystalline anisotropy. Both parameters decrease with increasing temperature. At low temperatures, these two effects extinguish and magnetic susceptibility remains nearly constant. If K decreases faster than M_S the thermal fluctuation at T close to T_C causes an increase in magnetic susceptibility, which is called the Hopkinson effect [16]. The increase in susceptibilities on the left-hand side of the susceptibility peaks in Fig. 3a–d is related to the pinning/unpinning of domain walls in pseudo-single- and multidomain material.

In order to investigate if this behavior has an effect on the field dependence, temperature dependence of magnetic susceptibility was measured in the temperature range −192 to 260 °C at three different field amplitudes (4, 50 and 300 A/m; Fig. 4a). While the curve shape of the 4 and 50 A/m measurements is similar with only slightly higher susceptibilities near the peak in the latter one, the 300 A/m measurement shows distinctly higher values over a significant temperature range. We interpolated susceptibility–temperature pairs with a resolution of 1 °C from the corrected raw data of Fig. 4a and calculated the field dependence parameter from the measurements at 300 and 50 A/m field amplitude (χ_{Hd} (%)$=[(k_{300A/m}-k_{50A/m})/k_{300A/m}]\times 100$). Fig. 4b illustrates a strong temperature dependence of the χ_{Hd} up to a maximum of ca. 34% in the temperature range between −100 and 260 °C. At room temperature, this sample shows χ_{Hd} of about 32%, which corresponds nearly to the value of 35% measured for this sample at room temperature in 30 and 300 A/m (see Table 1). This observation is significant either for the understanding of the increased field dependence parameter and for the strong scattering of χ_{Hd} in samples with intermediate titanomagnetite composition. The field dependence parameter changes over small temperature intervals, depending on the position within the curve. Therefore, the ambient measurement temperature has an influence on the calculated χ_{Hd} value.

Theoretical modeling for TM60 has shown that the blocking temperature of particles, which are domi-

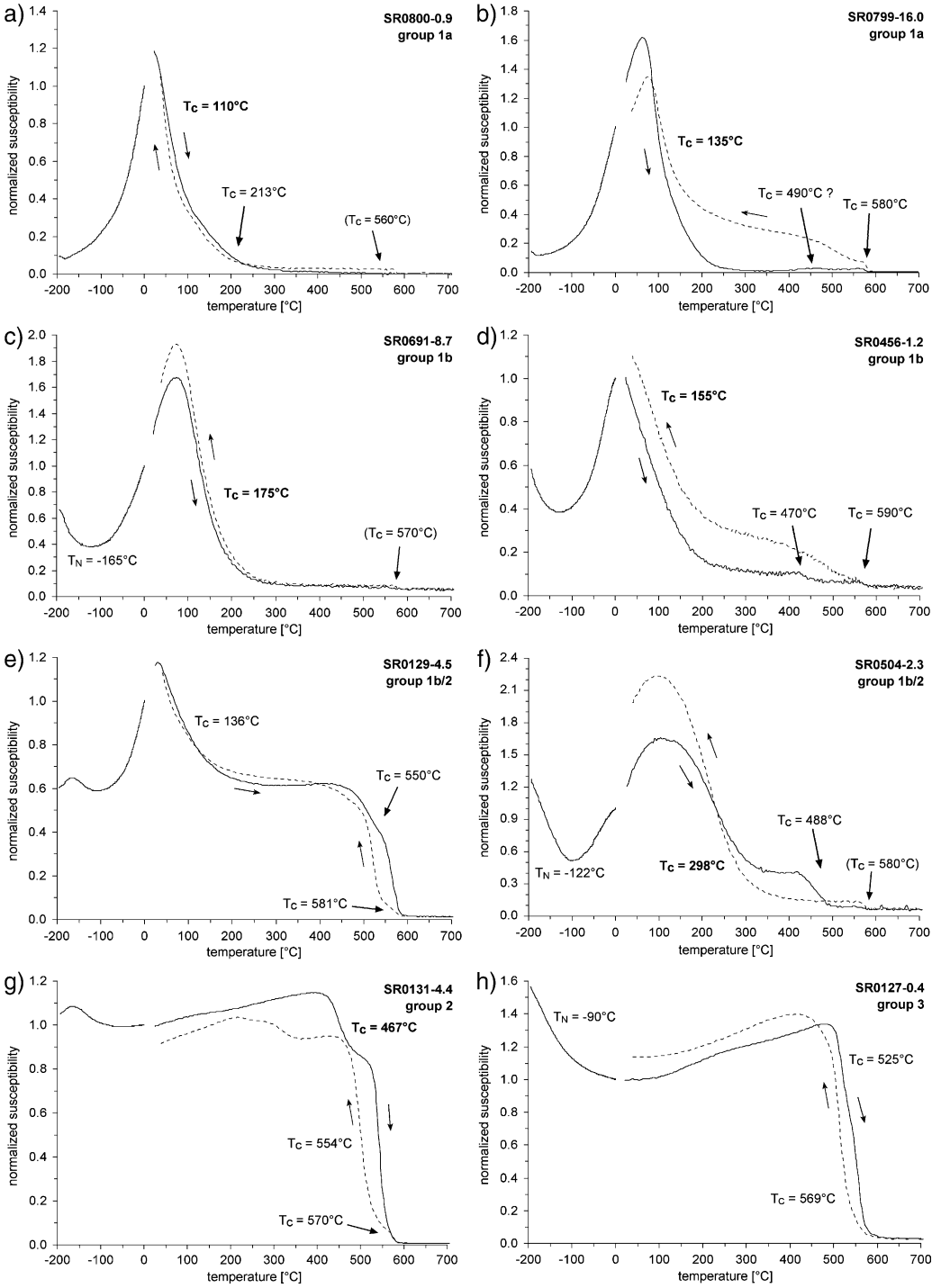

Fig. 3. Normalized susceptibility as a function of temperature between −192 and 700 °C. Full line is heating curve, dashed line is cooling curve (also indicated by small arrows), main Curie temperature is bold, minor ones are regular type letters.

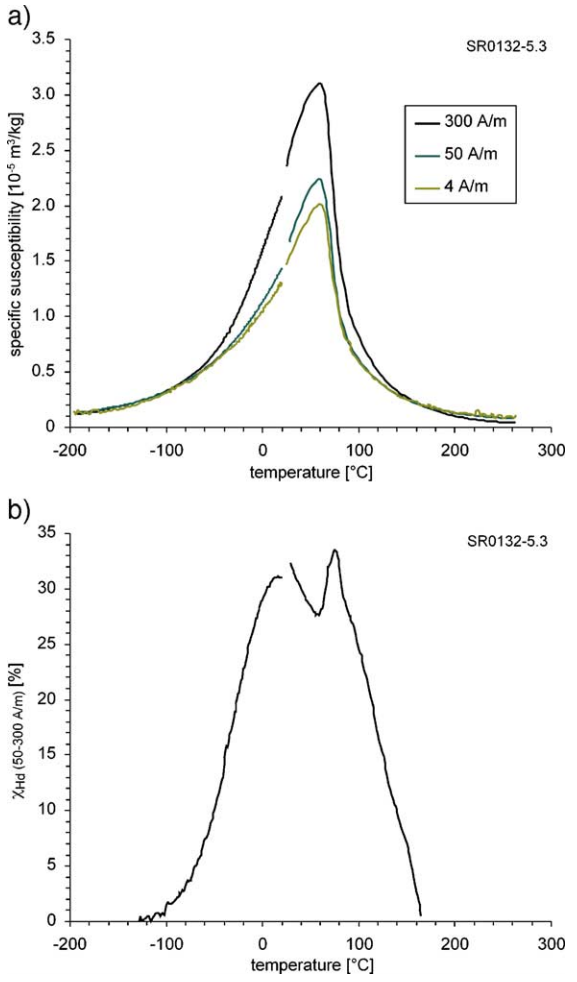

Fig. 4. (a) Specific susceptibility as a function of temperature between −192 and 260 °C at different field amplitudes and a constant frequency of 875 Hz. (b) Field dependence parameter χ_{Hd} (calculated for 50 and 300 A/m) as a function of temperature.

nated by magnetocrystalline anisotropy, is much lower than the Curie temperature [17]. Such particles are more sensitive for a change in magnetic domain configuration. Although Soffel and Appel [18] were not able to detect a different number of domain walls during IRM and TRM heating experiments of TM72 (X_{mt}=0.28), their study has shown a movement of domain walls. The movement of domain walls requires a higher wall displacement energy, which increases susceptibility.

Temperature dependent magnetic susceptibility measurements from M. Jackson and co-workers (http:/www.irm.umn.edu/bestiary/index.html) at different fields (30–2000 A/m) and frequencies (40–1000 Hz) have revealed that the characteristic peak of intermediate titanomagnetites shows nearly no frequency dependence. These data suggest that the differences in susceptibilities that we measured at different field amplitudes are actually related to the field amplitude and not to possible variations in the frequency. Hysteresis parameters from the HSDP-2 basalts have shown that grain sizes vary mainly in the single-, pseudo-single- and multidomain range (Fig. 5), which is in accordance with the assumption that superparamagnetic particles play a minor role. The increase of susceptibility with temperature for titanomagnetites with a peak-type $k(T)$ behavior is stronger at higher fields and is an important factor that has to be considered for the understanding of field dependence in intermediate titanomagnetites.

3.3. Effect of grain size

Worm et al. [3] have shown that the susceptibility of pyrrhotite and its field dependence strongly increases with grain size. While the susceptibility of grains smaller than 30 μm is field independent it increases for crystals larger than 100 μm in size in fields >10 A/m. Unpublished results of de Wall indicate that the field dependence of pyrrhotite is not only controlled by grain size but also by microstructures like, e.g. the defect concentration. Since the pyrrhotite samples from these studies consist of multidomain grains, the observed magnetization variations in low fields were interpreted from movement of domain

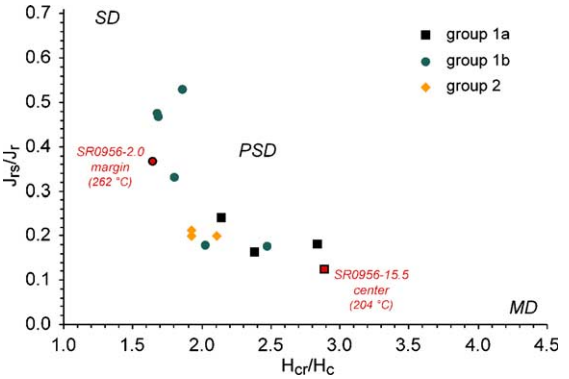

Fig. 5. Day plot shows the hysteresis parameter for some HSDP-2 basalts.

walls. A field dependence of susceptibility is also observed in pure hematite with the onset of multidomain behavior at grain sizes above 100 μm [4,5].

Wall movement is a major process in all multidomain materials at low fields and if the wall displacement energy is high enough a field dependence of susceptibility can occur. Therefore for each material, which shows field dependence, the grain size is a critical parameter. Fig. 6 shows the grain size variation (determined microscopically) of titanomagnetite with intermediate composition (groups 1a and 1b) versus the field dependence parameter. Curie temperatures are also given to estimate the influence of composition versus grain size. In general, samples with low T_C display the strongest field dependence as already shown in Figs. 1 and 2 and stated by Jackson et al. [6]. However the compositional effect seems to be accompanied by a grain size variation. Sample SR0456-1.2 shows a too small field dependence compared to the composition. In this sample, grain sizes only range up to 10 μm. Although the $k(T)$ curves display the occurrence of few Ti-poor titanomagnetite (Fig. 3d), the decrease of χ_{Hd} seems not to be connected with a dilution by these grains. This interpretation is drawn by comparing the similar $k(T)$ curves of sample SR0456-16.0 and SR0799-1.2 (Fig. 3b and d), which show similar composition and ratio of Ti-rich and Ti-poor titanomagnetite. The first one with larger grain sizes shows clearly a higher χ_{Hd} (Fig. 6a; Table 1). On the other hand, the field dependence parameter of sample SR0800-1.0 is distinctly larger than expected from its composition. The grain sizes of titanomagnetites from this sample range between 40 and 80 μm, but also grains of up to 160 μm occur.

In order to test if the observed microscopic grain size variations correspond to magnetic effective grain sizes, hysteresis loops were measured and J_{rs}/J_r was plotted versus H_{cr}/H_c (Fig. 5). The samples SR0956-15.5 and SR0956-2.0 originate from the central and marginal part of a submarine intrusion and are characterized by a different microscopic grain size range and slightly different composition (Fig. 6a; Table 1). In Fig. 5 they plot clearly on different positions with high H_{cr}/H_c and low J_{rs}/J_r ratios for the central sample and low H_{cr}/H_c and higher J_{rs}/J_r ratios for the marginal sample, indicating respectively larger and smaller magnetic grain sizes. Furthermore Fig. 6b shows a weak but positive correlation between field dependence parameter and H_{cr}/H_c ratio, indicating a larger average grain size for samples with stronger field dependence. These observations indicate that microscopically visible grain size and magnetically effective grain size have both some, although minor influence on the field dependence of titanomagnetite (Fig. 6a and b). Our observations confirm that small grain sizes of titanomagnetite (below about 10–20 μm) reduce and large grain sizes increase the field dependence of magnetic susceptibility in titanomagnetite-bearing samples, although we are aware that this effect is small compared to the composition/temperature effect.

3.4. Implications for AMS measurements in different basalt lithologies

The implication of field dependence of magnetic susceptibility in titanomagnetite for the anisotropy of magnetic susceptibility (AMS) has been mentioned in different studies [4,6,8]. Jackson et al. [6] already gave the advice to determine the AMS in field amplitudes of no more than a few tens of A/m to be sure that $M(H)$ is linear and the standard tensorial mathematics are valid. De Wall [8] showed for titanomagnetite-bearing rocks that the anisotropy factor (P') of the AMS ellipsoid is significantly increased at higher fields comparing measurements at 300 A/m and 30 A/m, especially for intermediate titanomagnetites with T_Cs between 128 and 160 °C, while the shape factor (T) and the orientation of the principal AMS axis appear to be insensitive to the field amplitude.

Using the commercial kappabridge KLY-2 of AGICO company AMS measurements were done at a field amplitude of 300 A/m and the shape of the AMS was determined for nearly 200 sample cylinders of different subaerial and submarine basaltic lithologies of the HSDP-2 borehole. The corrected degree of anisotropy ($P'=(\exp(2(\ln k_{max}-\ln k)^2+2(\ln k_{int}-\ln k)^2+2(\ln k_{min}-\ln k)^2)^{1/2})$, which describes the deviation from the spherical shape, and the shape factor of the ellipsoid ($T=(2\ln k_{int}-\ln k_{max}-\ln k_{min})/(\ln k_{max}-\ln k_{min})$), which provides information on the shape geometry of the ellipsoid (constrictional or flattening), are shown in Fig. 7. While the shape factor shows a significant variation in all lithologies, the degree of anisotropy is low with only minor scattering in the subaerial basalts and increases to higher values

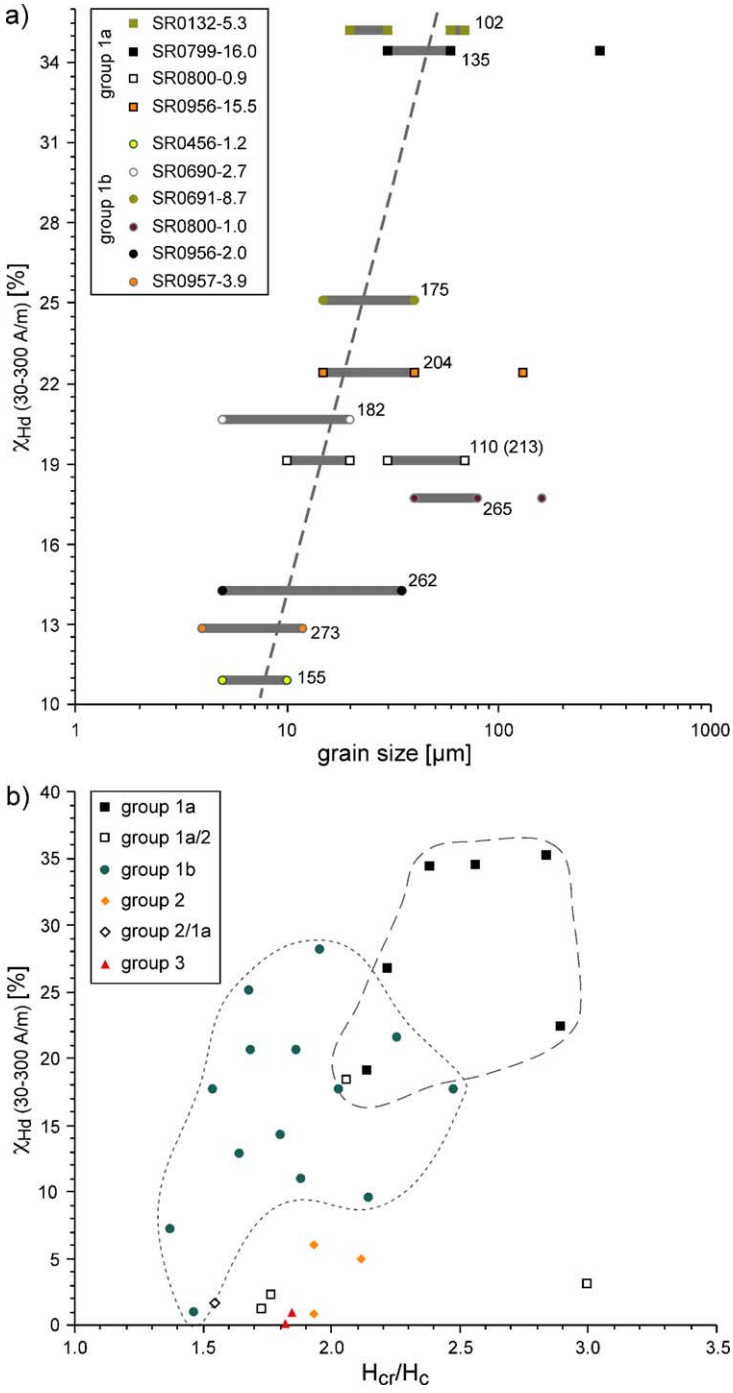

Fig. 6. (a) Field dependence parameter χ_{Hd} (calculated for 30 and 300 A/m) versus microscopic grain size range of titanomagnetite (numbers are titanomagnetite Curie temperatures); (b) χ_{Hd} versus H_{cr}/H_c ratio from hysteresis parameters as a measure of magnetic effective grain size.

with a strong scattering in the submarine lithologies. In the subaerial lithologies, oxyexsolution with hemoilmenite lamellae in Ti-poor titanomagnetite prevails. Since the crystallographically controlled hemoilmenite lamellae mostly occur along the (111)-planes in the titanomagnetite host, exsolution in titanomagnetite breaks down the degree of anisotropy caused by shape anisotropy. In the submarine lithologies, homogeneous titanomagnetite of intermediate compositions without exsolutions of hemoilmenite clearly dominate. Cañón-Tapia and Pinkerton [19] concluded from their experimental studies on basaltic Kilauea lava that the degree of anisotropy is a function of both, the thermal and shearing history of a sample. High degrees of anisotropy were found only in samples that were sheared at temperatures close to those encountered during eruption and then rapidly quenched. Such a cooling history is also assumed for the massive basalt, pillow and intrusion units of the submarine part of the Mauna Kea from this study [10]. Cañón-Tapia and Pinkerton [19] describe low degrees of anisotropy if samples are allowed to cool down slowly without further deformation. This is in good agreement with our findings of low degrees of anisotropy in the sub-

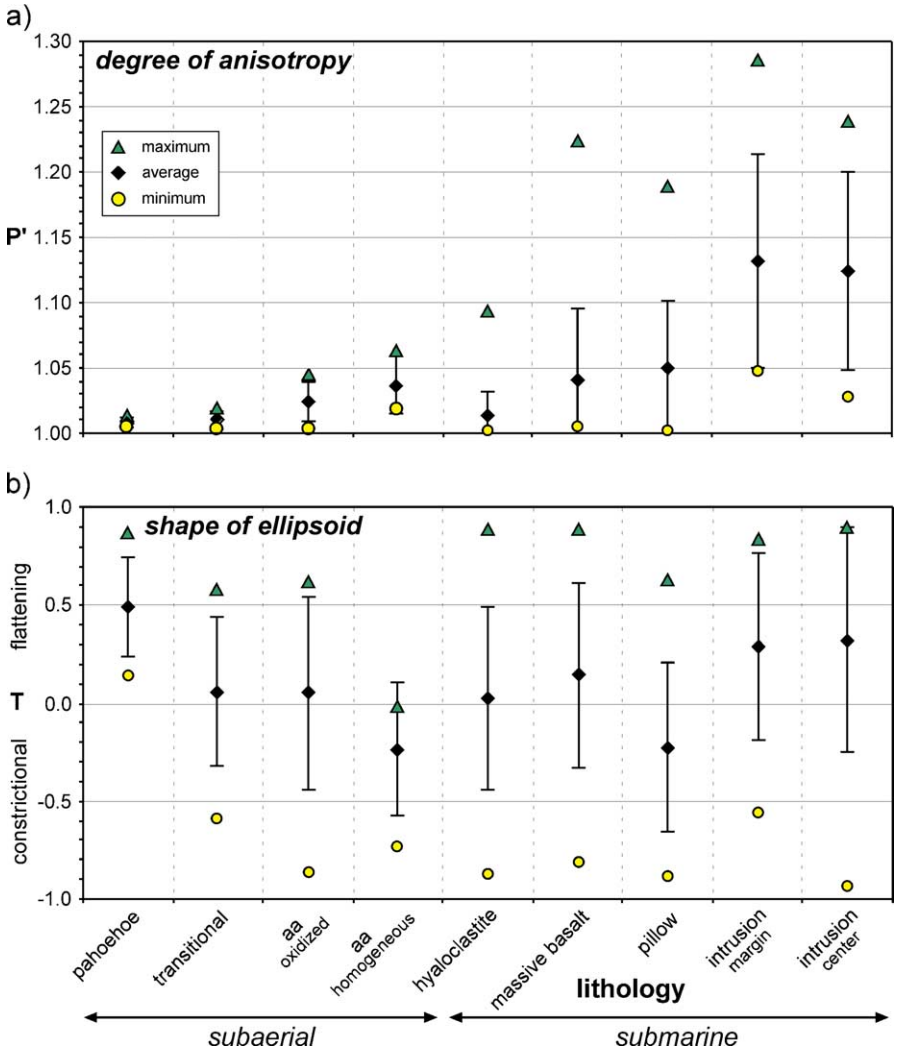

Fig. 7. (a) Degree of anisotropy (P') and (b) shape factor (T) of different subaerial and submarine basalt lithologies.

aerial lithologies, which are strongly influenced by high-temperature oxidation during cooling and therefore are dominated by sub-solidus reactions creating Ti-poor titanomagnetites and exsolved hemoilmenite lamellae (compare Fig. 5d in [10]). We assume that this texture is the main reason for the strong decrease of the degree of anisotropy. Cañón-Tapia and Pinkerton [19] found no reason to suspect an important contribution from the compositional factor and concluded that composition and microscopic characteristics of ferromagnetic minerals have only a minor influence on the relation between AMS and lava shearing. This is in contrast to the conclusions of de Wall [8] and our own observations, which indicate a strong influence of composition on the degree of anisotropy for intermediate titanomagnetites.

Fig. 8 shows the Jelinek diagram for selected samples of the HSDP-2 basalts with shape factor T plotted versus corrected degree of anisotropy (P') calculated from AMS measurements at fields between 2 and 450 A/m. Samples with a low field dependent parameter (e.g. SR0131-4.4) show only little variation in the P' at different field amplitudes, while samples with a stronger field dependence (e.g. SR0799-16.0) show a variation up to about 4% of the degree of anisotropy for the investigated samples. This behavior confirms the correlation between titanomagnetite composition, field dependence and degree of anisotropy, which was already described by de Wall [8]. Furthermore, our data suggest that samples, which show a high degree of anisotropy already at low fields in combination with a high field dependence parameter (e.g. SR0691-8.7), show the highest variation of P' for the different fields. In this case, the deviation of P' between the measurement at 6 A/m and 450 A/m ($\Delta P'$) can reach about 9%.

Fig. 9a shows the field dependent parameter versus $\Delta P'$ calculated from the 450 and 6 A/m measurement. In general a positive trend between these parameters can be observed. Group 1a samples show a high χ_{Hd} and a medium $\Delta P'$ indicating a steep slope while samples of group 1b show higher $\Delta P'$ values at similar χ_{Hd} suggesting a less steep slope. Plotting P' at 6 A/m versus $\Delta P'$, a positive correlation is found especially for group 1b samples above a critical degree of anisotropy of 2–3% (Fig. 9b). In a 6 A/m field the relationship between magnetization and magnetizing field is linear, and P' represents the true linear tensorial AMS, which we refer to as the extrinsic anisotropy of the sample. The extrinsic anisotropy depends on the shape and/or distribution anisotropy of grains and is controlled in the HSDP-2 basalts by viscosity and quenching/crystallization history of the lava. $\Delta P'$ is influenced by the deviation arising from

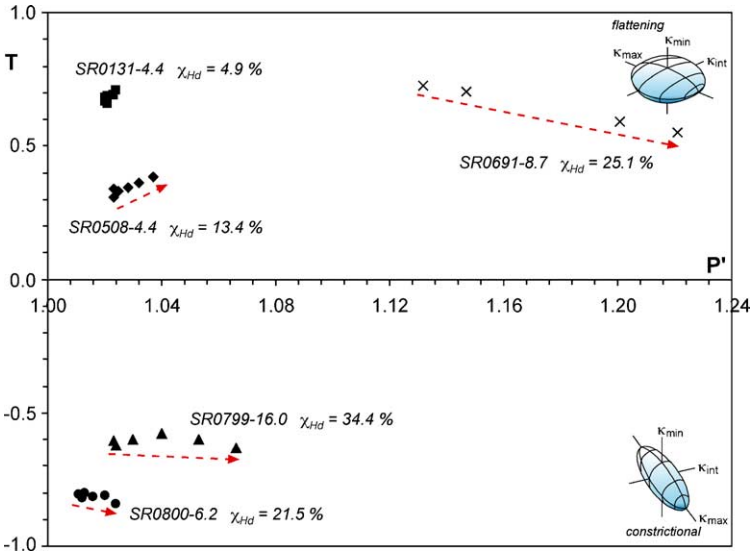

Fig. 8. Jelinek diagram showing shape factor (T) versus degree of anisotropy (P') for selected samples, where AMS was measured in fields between 2 and 400 A/m (6, 50, 200 and 450 A/m for SR0691-8.7).

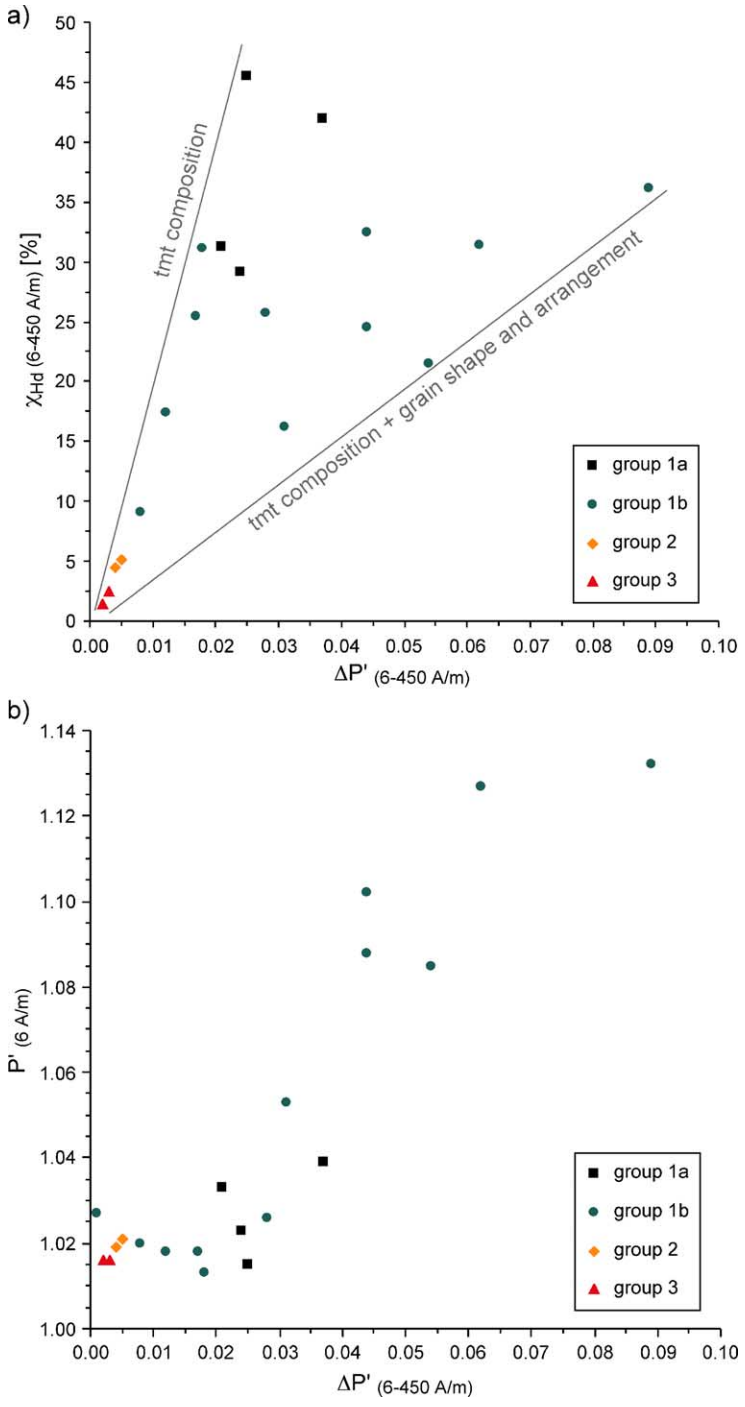

Fig. 9. (a) Field dependence parameter χ_{Hd} versus $\Delta P'$ calculated from the 6 and 450 A/m measurements for different titanomagnetite types from the HSDP-2 cores; (b) degree of anisotropy (P') at 6 A/m versus difference of P' at 450 and 6 A/m ($\Delta P'$).

the linear to the non-linear relationship between magnetization and magnetizing field, which is related to the magnetocrystalline, intrinsic anisotropy of the sample. These observations suggest that the field dependence effect on $\Delta P'$, which is primarily caused by titanomagnetite composition, can be enhanced by an anisotropy effect.

3.5. Effect of arrangement between the rhombohedral and cubic Fe–Ti oxides

The corrected degree of anisotropy (P') is mostly used instead of the degree of anisotropy (P) because it characterizes the quantity of the anisotropy somewhat better than P [20]. But from the more simple formula $P=k_{max}/k_{min}$ it can be easily seen that this factor describes the aspect ratio of the principal susceptibility axes. For non-spherical TM40 and TM60 specimens, the demagnetizing factor N was found to be significantly less than 1/3 measured along their long dimensions [6]. This behavior indicates that for elongate specimen, self-demagnetizing limits are not attained and the magnetic susceptibility continuously increases in the applied field. The strong field dependence of P' for intermediate titanomagnetites and the lower self-demagnetization factor for anisotropic samples (caused by elongated shapes or preferred arrangement of grains) imply that the increase of magnetic susceptibility is stronger in the direction of k_{max} than k_{min}.

In order to test this hypothesis, the magnetic susceptibility at different field amplitudes was measured for anisometric polycrystalline synthetic samples consisting of titanomagnetite (T_Cs ranging between 30 and 160 °C) and hemoilmenite parallel and orthogonal to their long dimension (Fig. 10). The increase in susceptibility with field amplitude is significantly stronger parallel to the long dimension than orthogonal. This behavior is especially significant in samples where the titanomagnetite grains form elongated aggregates separated by hemoilmenite (Fig. 10b) but can also be observed in samples, which contain isometrically grains of nearly equal amounts of both phases (Fig. 10c).

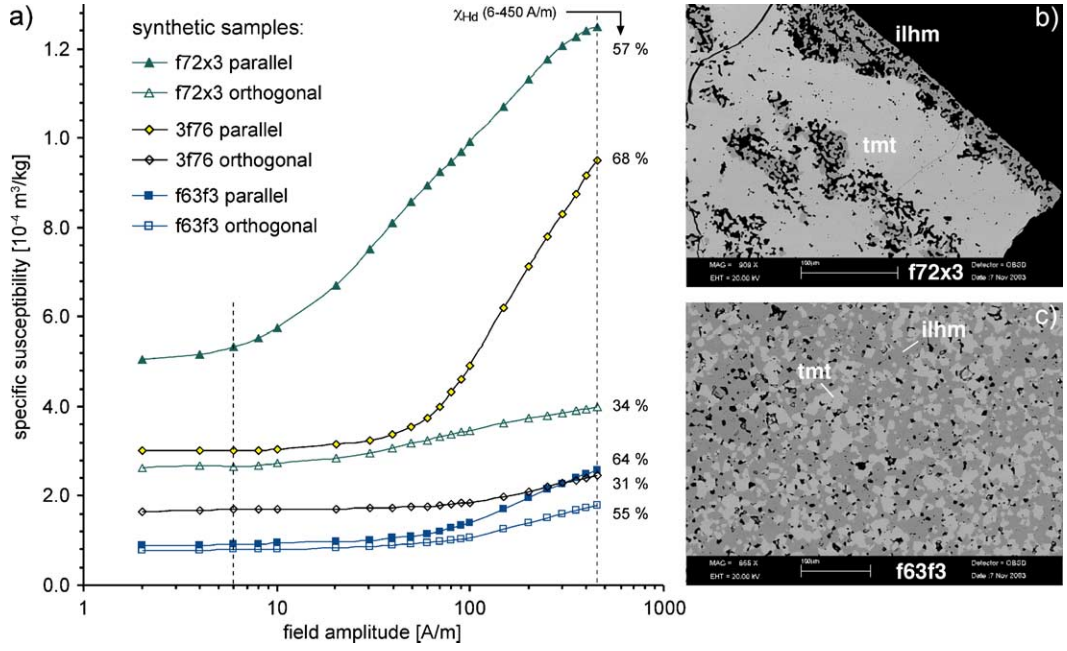

Fig. 10. (a) Specific susceptibility as a function of field amplitude for synthetic samples consisting of titanomagnetite and hemoilmenite (numbers at curve are χ_{Hd} calculated from measurements at 6 and 450 A/m, see dashed lines); (b) backscatter electron image of synthetic sample f72x3 and of f63f3 (c) showing arrangement of titanomagnetite (tmt) and hemoilmenite (ilhm; BSE images were provided by R. Engelmann, Heidelberg).

Microscopic observations of the HSDP-2 basalt samples have shown that especially group 1b samples with intermediate titanomagnetites contain elongated dendritic to skeletal oxide grain morphologies typical for rapid cooling rates [21]. Often, hemoilmenite crystallizes first forming elongated dendrites, which act as nucleus for the titanomagnetite grains (Fig. 11). Furthermore, titanomagnetite crystals show a chain-like arrangement. Especially these samples exhibit high degrees of anisotropy already at low fields of 6 A/m and are found in the massive basalt, pillow and intrusion units of the submarine stage (Fig. 7). It is also these samples, which show the linear P' versus $\Delta P'$ behavior in Fig. 9b. Field dependent susceptibility measurements parallel to k_{max} and k_{min} of sample SR0691-8.7, which shows a high extrinsic P' value of 1.13 at 6 A/m, revealed a difference in the χ_{Hd} parameter of 4%. Although this is much less compared to the synthetic samples, which show differences in the χ_{Hd} values perpendicular to each other between 9 and 37%, there is a clear indication that the field dependence of P' for submarine basalts containing intermediate titanomagnetite and hemoilmenite is strongly influenced by its grain arrangement. Our study shows that cooling history and the resulting magneto-mineralogy is a very important factor, which controls the degree of anisotropy in different basaltic lithologies (Fig. 7).

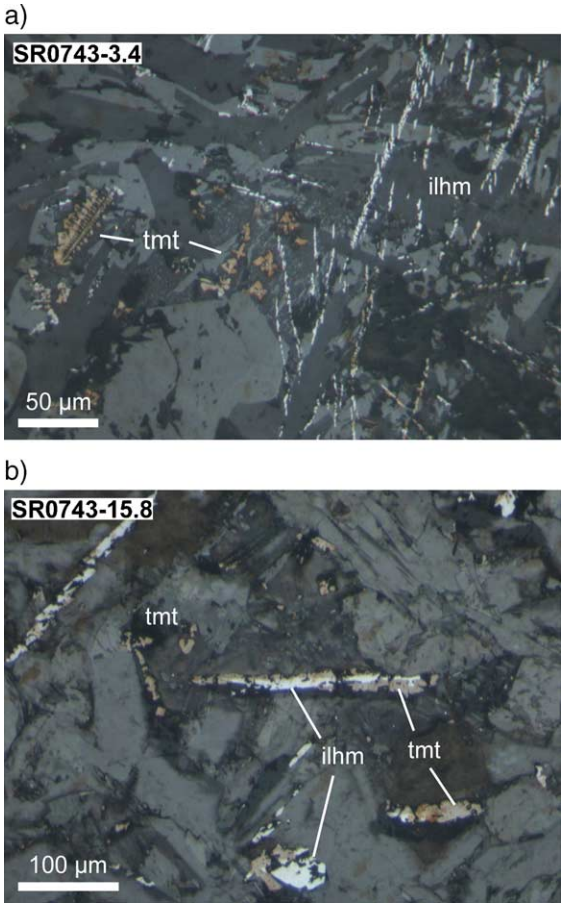

Fig. 11. Thin section images with Fe–Ti oxide textures (coated with ferrofluid and in oil immersion) of group 1b samples SR0743-3.4 (a) and SR0743-15.8 (b), which show high degrees of anisotropy (tmt: titanomagnetite, ilhm: hemoilmenite).

4. Discussion

Field dependent magnetic susceptibility measurements of basalts from the HSDP-2 borehole, Hawaii, confirmed the generally described correlation between field dependence and titanomagnetite composition [6] but revealed also a significant scattering, especially for intermediate titanomagnetites with T_C between 100 and 300 °C. This is in contrast to studies of de Wall [8] and de Wall and Nano [15], which showed a good correlation with only slight scattering between field dependence parameter and titanomagnetite Curie temperature (compare Fig. 1) for mafic alkaline volcanic rocks from the Tertiary volcanic province in SW Germany (olivine-melilititic dike of Hegau and subaerial Ti-basanite of Vogelsberg). These rocks contain Mg–Al–(Cr-)bearing titanomagnetites but no hemoilmenite.

A detailed study on HSDP-2 basalts examining in addition to composition the effect of temperature, grain size and anisotropy on the field dependent susceptibility revealed that all these parameters have an influence. The least significant impact is that of grain size, the effect of titanomagnetite composition is strongest. The titanomagnetites from the HSDP-2 basalts contain low Mg (0.01–0.19 per formula unit, and up to 0.22 in rims around chromian spinel) and low Al (0.04–0.13 per formula unit, and up to 0.17 in rims around chromian spinel [10]) compared to higher values (Mg: 0.11–0.39, Al: 0.08–0.24 per formula unit [14]) for the mafic alkaline volcanics from de Wall [8]. According to Soffel et al. [22] the domain structure of TM60 and TM60 contaminated with Al, Mg,

Mn and V is similar. But, the substitution of Fe, especially by Al, shifts the Curie temperature to lower values [23] and also intrinsic properties like the magnetostriction are modified [24]. The higher amount of foreign ions in titanomagnetite from the mafic alkaline volcanics and the resulting lower T_C could explain why the χ_{Hd} data from the mafic alkaline volcanics describe the lower limit of the HSDP-2 data set (compare Fig. 1). Besides these compositional differences the HSDP-2 basalts show higher degrees of anisotropy ranging from 1.01 to 1.27 (measured at 300 A/m; at 6 A/m: $P' < 1.16$) in comparison to lower P' of maximal 1.14 at a field amplitude of 300 A/m and up to 1.08 for 30 A/m for the alkaline volcanics [8,14].

One reason for the scattering observed in Fig. 1 is the behavior of susceptibility as a function of temperature, which shows for the intermediate titanomagnetites the so-called peak-type behavior (Fig. 3a–d). Although the curves obtained from $k(T)$ measurements at different field amplitudes indicated that the geometry of the curve defined by the full width at half maximum remains constant (which is important for consistent T_C determinations), the absolute susceptibility is much larger in the 300 compared to the 50 A/m measurement. Calculation of a field dependence parameter between $k(T)$ curves at 50 and 300 A/m yielded significant changes of the field dependence over the temperature interval −100 and 260 °C (Fig. 4b). The increase of susceptibility is an indication of the movement of domain walls involving a higher wall displacement energy. This feature is characteristic for intermediate titanomagnetites, but can cause problems for the use of χ_{HD} measured at ambient temperatures. Small temperature changes during measurement and differences in peak-shape for different samples could be the reason for some scattering in χ_{HD} parameter (see Fig. 4b).

Grain size effects of the titanomagnetite crystals can also contribute to the scattering, because of changes in the balance of different energies. Magnetostatic energy is larger for grains with fewer domains, i.e. grains with smaller grain sizes. In smaller grains magnetostatic energy dominates, whereas in larger grains the magnetostatic energy is reduced by a higher amount of domain walls resulting in a higher domain wall energy. This change in dominating energies enables domain wall movements and increases the measured susceptibility at higher fields. Therefore, with respect to the titanomagnetite composition smaller grains have a reduced field dependence parameter compared to larger grains with the same composition. Our results suggest that a weak grain size effect in titanomagnetite occurs at grain sizes of a few to several tens of μm (Fig. 6a), which is well above the critical single-domain size limit of 0.6 μm, reported by Soffel [25] for natural TM60. From his investigations and from theoretical domain state calculations for TM60 [17] multidomain behavior (more than 8 domains) can be expected for grains larger than ca. 15–40 μm, which is in good agreement with our own observations from e.g. scanning electron microscopy and hysteresis measurements. Probably, larger titanomagnetite grains than observed in our study cause a much clearer field dependence. For hematite Kletetschka and Wasilewski [5] found a weak field dependent behavior for multidomain grains of 100 μm size and a significant field dependence for grains larger than 200 μm (whereas the critical single-domain size for equidimensional particles is 15 μm [26]). After Soffel [27] multidomain behavior for pyrrhotite should occur for grain sizes above ca. 20 μm. This agrees almost perfect with the study of Worm et al. [3], who found field dependent susceptibility for grains larger ca. 25 μm, whereas grains above 100 μm show a clear field dependence with a strong increase of susceptibility at higher field amplitudes. Hematite is absent in almost all HSDP-2 basalts, except in highly oxidized subaerial basalt lava of sample group 2 or 3, which show nearly no field dependence. Pyrrhotite, which has been found in some submarine HSDP-2 basalts as a subordinate opaque phase, shows grain sizes of less than 40 μm and therefore does not contribute to the field dependence of the HSDP-2 basalts. Furthermore, the amount of pyrrhotite is very low compared to the Fe–Ti oxides, and therefore the influence of this mineral on the magnetic properties of the basalts is negligible.

In the group 1b samples, especially from the submarine HSDP-2 basalt lithologies, the rapid quenching of rhombohedral and cubic oxides and the missing of strong oxidation preserved a higher degree of anisotropy compared to the subaerial basalts (Fig. 7). In these samples the field dependence parameter seems to be additionally modified according to the direc-

tional dependence of the χ_{Hd} parameter on the AMS texture (parallel k_{max} or k_{min} axis). This conclusion can be drawn from field dependent measurements of the natural basalt samples in different sample orientations as well as of the synthetic anisometric grain aggregates (Fig. 10). Furthermore, this mechanism is reflected by the variation of P' derived from AMS measurements at different field amplitudes. Due to this behavior the field dependence parameter can vary by several percents, comparing measurements in k_{max} or k_{min} directions. Therefore, additionally to the temperature dependence of the χ_{Hd} parameter and the subordinate grain size effect the magnetic fabric has to be considered for field dependent susceptibility measurements and for the usage of a field dependence parameter (at room temperature) as proposed by de Wall [8].

Jackson et al. [6] suggested for measurements of the anisotropy of susceptibility in titanomagnetite-bearing rocks field amplitudes less than a few tens of A/m to assure a linear relationship between magnetization and the magnetizing field. Commercial kappabridges (e.g. KLY-2 of AGICO) run at field amplitude of 300 A/m and only with the new generation of instruments (e.g. KLY-4S) an adjustment of the field amplitude in the interval between 2 and 450 A/m is possible. Hrouda [4] pointed out that the field dependence problem has not only be considered for titanomagnetite-bearing rocks but also for hematite- and pyrrhotite-bearing ones. For rocks with pure magnetite as well as dia- and paramagnetic rocks susceptibility is field independent.

For the investigated HSDP-2 basalts the degree of anisotropy (P') at weak fields is caused by the grain shape and alignment (extrinsic factors) related to the viscosity and quenching history of the lava. The increase of P' ($\Delta P'$) at higher field amplitudes is controlled by intrinsic factors like the Ti-content, magnetocrystalline anisotropy, defects and internal stresses of titanomagnetite. For samples with similar composition $\Delta P'$ is larger, if P' is already high at low fields. The degree of anisotropy is also controlled by the magnetostatic interactions between the particles, because the grains are not only influenced by the external magnetic field (H_a) but also by dipole fields created by the particles themselves [28]. The demagnetizing energy in direction of the strong interactions is reduced, which enhances the wall displacement energy. This process is responsible for the stronger increase in susceptibility parallel to k_{max} direction. The demagnetizing factor N decreases significantly for increasing aspect ratios (e.g. aspect ratio (length/width)=0.2, N=0.751 and aspect ratio=4, N=0.075 [29]), and according to $H_{eff}=H_a-NM$, this behavior means that the effective magnetic field (H_{eff}) is significantly lower perpendicular to k_{max} than parallel to it. These results indicate that directional field dependent measurements of magnetic susceptibility may provide information about the magnetic microstructure of titanomagnetite.

5. Conclusions

The dependence of AC susceptibility on the applied field amplitude was shown previously (e.g. [6,8]) to correlate strongly with Ti-content of titanomagnetite grains. The application of the χ_{Hd} parameter (χ_{Hd} (%)=$[(k_{300\ A/m}-k_{30\ A/m})/k_{300\ A/m}]\times 100$ [8]) on the HSDP-2 samples revealed a significant scattering, especially for intermediate titanomagnetite compositions of groups 1a and 1b (Figs. 1 and 3). Our study has shown that the field dependence of magnetic susceptibility in the titanomagnetite-bearing basalts of the HSDP-2 borehole from Hawaii is controlled by multiple factors, which can overlap and impede the interpretation of such data in terms of composition.

In the subaerial HSDP-2 basalts, high-temperature oxidation often changed the primary composition of titanomagnetite towards Ti-poorer compositions. Submarine units are characterized by homogeneous titanomagnetite mainly of intermediate compositions, which are associated with small amounts of Ti-poor titanomagnetite and/or titanomaghemite. These samples show the strongest scattering of the χ_{Hd} parameter and were investigated in more detail in this study.

Additional to the composition and mixing of titanomagnetite of different composition, the temperature dependence of the χ_{Hd} parameter around room temperature (Fig. 4), grain size (Fig. 6) and, more important, the magnetic fabric (Fig. 9, 10) plays an important role.

Therefore, this study shows that the interpretation of AMS data from basaltic rocks requires a good

understanding of the magneto-petrology, especially if intermediate titanomagnetite is present. Intrinsic (Ti-content, magnetocrystalline anisotropy) and extrinsic (shape and alignment of grains) factors control the degree of anisotropy and must be considered when interpreting AMS data in terms of strain rates experienced by moving lava during emplacement.

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